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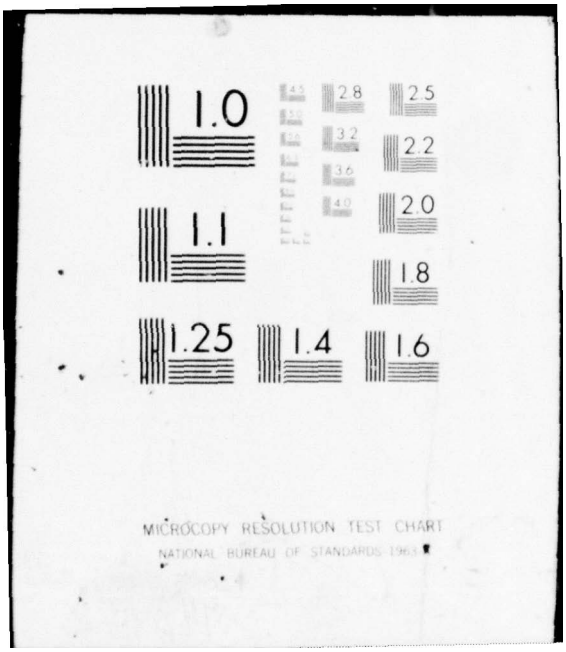
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AFGL-TR-76-0161
ENVIRONMENTAL RESEARCH PAPERS, NO. 574

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Cryogenic, Whole-Air Sampler and Program for Stratospheric Composition Studies

CHARLES C. GALLAGHER
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20 July 1976

AERONOMY DIVISION PROJECT 6687
AIR FORCE GEOPHYSICS LABORATORY
HANSCOM AFB, MASSACHUSETTS 01731

AIR FORCE SYSTEMS COMMAND, USAF

This technical report has been reviewed and is approved for publication.

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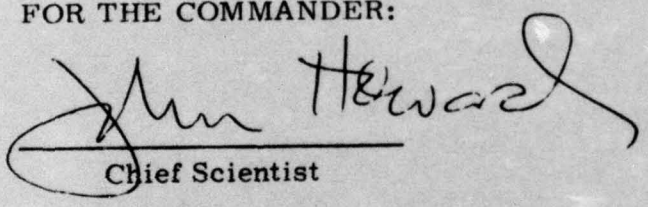

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REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: AFGL-TR-76-0161

2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle): CRYOGENIC, WHOLE-AIR SAMPLER AND PROGRAM FOR STRATOSPHERIC COMPOSITION STUDIES

5. TYPE OF REPORT & PERIOD COVERED: Scientific. Interim.

6. PERFORMING ORG. REPORT NUMBER: ERP No. 574

7. AUTHOR(s): Charles C. Gallagher
Robert V. Pieri, Capt, USAF

8. CONTRACT OR GRANT NUMBER(s)

9. PERFORMING ORGANIZATION NAME AND ADDRESS: Air Force Geophysics Laboratory (LKD)
Hanscom AFB
Massachusetts 01731

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: 62101F
66870301

11. CONTROLLING OFFICE NAME AND ADDRESS: Air Force Geophysics Laboratory (LKD)
Hanscom AFB
Massachusetts 01731

12. REPORT DATE: 20 July 1976

13. NUMBER OF PAGES: 20

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)

15. SECURITY CLASS. (of this report): Unclassified

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

AFGL-TR-76-0161, AFGL-ERP-574

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Cryogenic, whole-air sampler
Stratospheric composition

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A program has been initiated to measure the stratospheric content of NO, NO_2, CO, SO_2, CH_4, NH_3 and other trace gases which may influence our environment. The initial or baseline concentrations of these gases are being measured as a function of altitude and latitude. Subsequent measurements will indicate any time dependency in these concentrations and permit a possible correlation with increased Air Force stratospheric operations. Latitudes from Panama to Alaska and altitudes from 12 to 30 km will be investigated.

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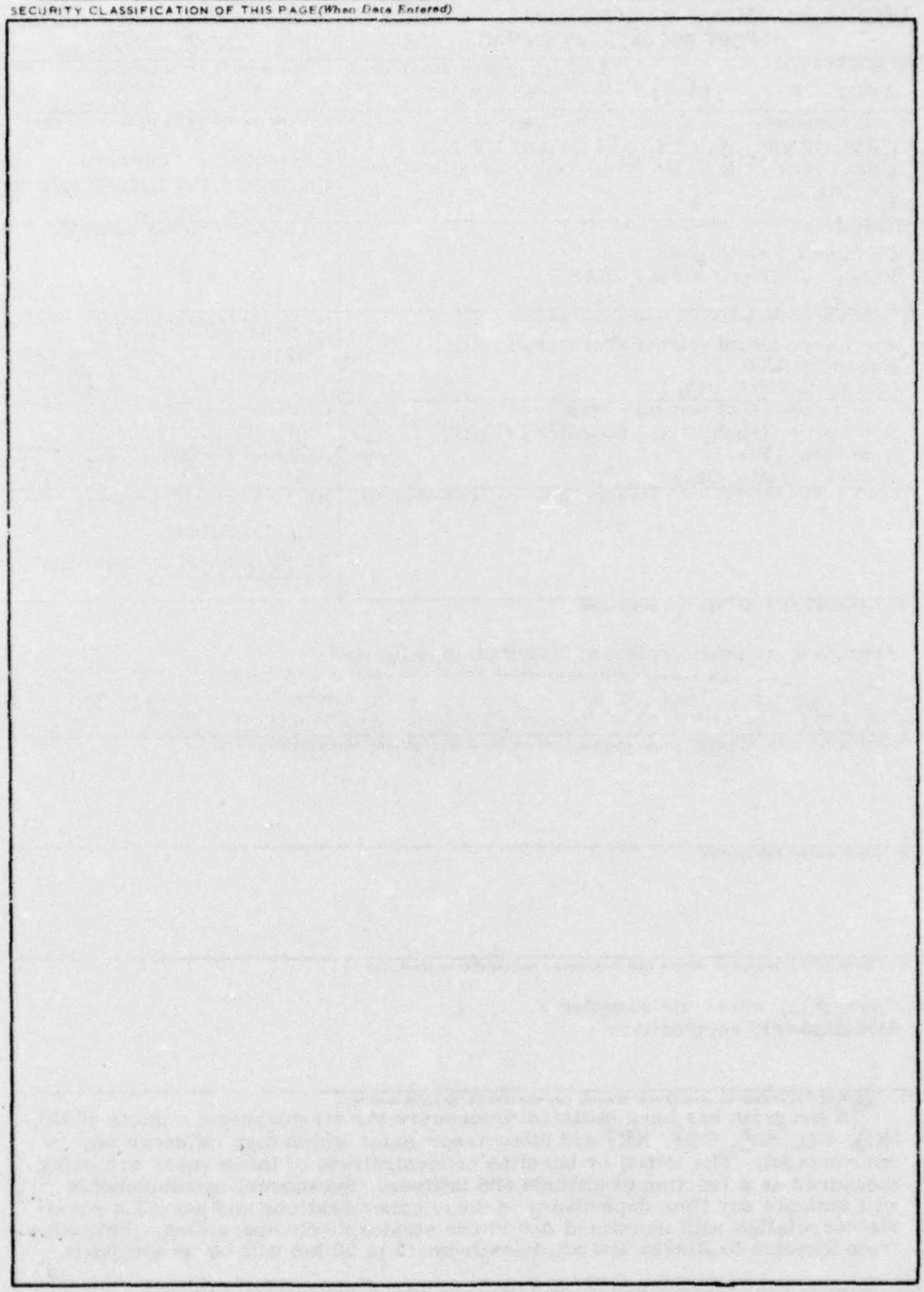
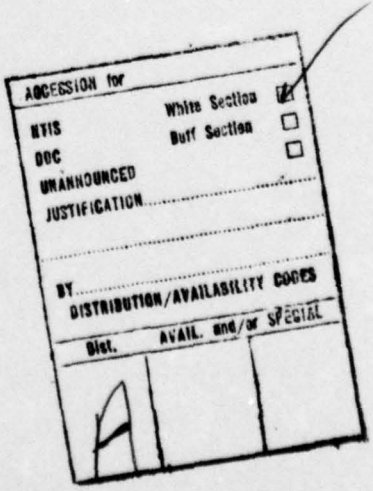


Preface

The authors wish to thank Rocco S. Narcisi for initiating the study and for his continued assistance and encouragement, Joseph M. Calo for initial design work including that on the whole-air sampler, Hans Laping for designing and supervising operation of the electronic command and monitoring system, and S Sgt Anthony Coriaty, Frederick Dale, and Gennaro S. Federico for their generous assistance.



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1. INTRODUCTION

The stratosphere, that portion of the atmosphere extending from the tropopause to about 50 km in altitude, plays a significant role in protecting our environment. Various trace gases such as CO, H_2O, $CFCl_3$, CF_2Cl_2, and other hydrocarbons play an important role in the physical chemistry and photochemistry of the stratosphere although they are present only in parts per million to parts per billion and lower concentrations. Stratospheric ozone, for example, absorbs over 99% of the harmful solar ultraviolet radiation that would otherwise impinge on the earth's surface even though the peak O_3 concentration is only a few parts per million. The situation is further complicated by the fact that the stratosphere has a long residence time for contaminants.

The injection of gaseous contaminants into the stratosphere can lead to significant but hard to predict worldwide climatological and biological effects by disturbing the delicate balance that exists, and thus, it is imperative to periodically monitor the concentrations of these minor species as well as to investigate their sources and sinks.

Of particular interest to the Air Force is the determination of any environmental effects of the B-1, F-15, and other Air Force stratospheric operations. To this end, a program has commenced with the goal of measuring, first, the

(Received for publication 19 July 1976)

natural or baseline levels of the minor species and then their variations in time and space as Air Force stratospheric operations expand. Also, the mixing and interaction of jet exhaust species with atmospheric constituents will be measured. The results will yield recommended standards for Air Force operations and an assessment of the environmental impact of the B-1, F-15, and future Air Force aircraft.

2. APPROACH TO STRATOSPHERIC COMPOSITION MEASUREMENTS

2.1 Altitude and Latitude

The mixing ratios of the major atmospheric constituents remain about the same throughout the troposphere and stratosphere. The minor species, however, have been found to exhibit altitude variations in their mixing ratios, but the available data is limited and quite variable.

Therefore, measurements should be obtained at various altitudes within the stratosphere and, particularly, its lower region. It is at these lower stratospheric altitudes where Air Force operations should be more numerous and also where many of the mixing ratios and most of the rates of change of these ratios are expected to be the greatest for the trace gases of interest.

The altitudes chosen for investigation are 12, 15, 20, 25, and 30 km.

Various physical and chemical factors which contribute to the aeronomy of the stratosphere result in the mixing ratios of the minor species varying with latitude. For this reason, measurements will be obtained at five latitudes in the northern hemisphere, specifically: Panama, Alaska, northern California, southern New Mexico, and a yet to be determined location near the U. S. -Canadian border. The higher concentration of measurements at midlatitudes is consistent with the latitude range of the more heavily used flight paths.

2.2 Temporal Variations

The initial measurements obtained at each altitude and latitude will be considered to be the natural or baseline values. Succeeding measurements, obtained periodically over a span of at least five years, will reflect any changes with time of the mixing ratios of the minor species. Such changes could then be correlated with increases in stratospheric flight operations.

It should be noted, however, that many measurements are needed to determine true year to year changes as one specific measurement can be affected by local stratospheric inhomogeneities.

2.3 Why a Cryogenic Whole-Air Sampler?

It was decided that the air collection procedure would utilize cryogenic methods. A major benefit of this would be increasing the quantity of trace gases obtained through the collection of a large sample. The other major benefit would be that any highly reactive constituents which might be in the stratosphere would be trapped, by freezing, before they could undergo reactions. Thus, the chances of metastable compounds remaining in their natural concentrations long enough to allow precise laboratory analysis would be considerably enhanced.

Since there is a direct relationship between reaction rate and temperature and since several of the reactions of interest involve nitrogen (N.B.P. = 77^{o}K), it was decided to use liquid helium because of its low boiling point (N.B.P. = 4^{o}K), the past experience in using it, the lower cost and, finally, because it is easier to obtain in the field.

2.4 Why Balloons?

The altitudes of interest are from 12 to 30 km. The practical work ceiling for most aircraft is less than 18 km and rockets and satellites are most useful above 40 km; thus, it seemed that a balloon platform was the logical choice. Also, a large research balloon is finely controllable in its vertical flight, thus allowing more precise construction of altitude profiles. The flexibility in choosing vertical motion also permits the positioning of the balloon at a given altitude following sampling, to take advantage of the wind speed and direction at that altitude to carry the balloon over a favorable location for termination and recovery.

3. WHAT DO WE HOPE TO MEASURE?

Each sample to be collected consists of 1 mole (22.4 liter) of whole air. A large sample such as this provides an excellent opportunity to detect all those gases present in the sample at mixing ratios down to one part per billion or lower.

In this way, the known stratospheric constituents and those present but not previously detected can both be measured with a high degree of accuracy.

Current interest in the stratosphere focuses on the destruction of ozone resulting from the diffusion into this region of the chlorofluoromethanes used both in refrigeration and as the propellant in aerosol cans. Jet engine effluents such as nitric oxide (NO) and nitrogen dioxide (NO_2) also result in the destruction of ozone. For this reason and, because of their relation to Air Force operations, these two gases will be of prime interest in the stratospheric composition program. Naturally, the chlorofluoromethane content will also be investigated.

Other gases, some of which are included in jet engine effluents, play an important role in the physical chemistry and photochemistry of the stratosphere and will be studied. These include: sulfur dioxide (SO_2), sulfur trioxide (SO_3), carbon monoxide (CO), ammonia (NH_3), methane (CH_4), carbon dioxide (CO_2), nitrous oxide (N_2O), and nitric acid (HNO_3).

4. DESIGN AND CONSTRUCTION OF THE CRYOGENIC AIR SAMPLER

The basic design of the sampler is shown in Figure 1. The sampler collects one mole of gas through a viscous leak in approximately one hour, by condensation on the cold wall. The total cryogenic hold time of the sampler is 20 hours using liquid helium; however, the hold time can be increased to 70 hours by using liquid neon (NBP = 27°K). Use of liquid neon would sacrifice the pumping of helium and hydrogen gas, however. As a required industry safety feature, a fracturable metal diaphragm is connected to the sample container. The particular diaphragm utilized ruptures when the interior pressure rises above 18 psia, saving the internal parts from damage if the sample should be inadvertently warmed.

The sampler cylinder is surrounded by the primary cryogen which is surrounded with a portion of the thermal guard vacuum (5×10^{-4} Torr) then a liquid nitrogen heat shield, then another section of the guard vacuum, and finally the external wall. Precautions are taken to ensure the integrity of the gas collected, for example, gold plating on most of the interior surface, pre-flight evacuation to 10^{-8} Torr pressure while heating to over 350°C, and so forth.

A more thorough treatment of the internal structure can be found elsewhere.[1]

The viscous leak is welded to a double sided vacuum flange, allowing it to be changed for various sampling altitudes. The leak used at 20 km is an 18.4-cm long tube with an inside diameter of 0.15 cm. Using Poiseuille's Equation,[2] the leak rate of ambient air through this tube is predicted to be approximately 7.4 standard cm^3 of gas per second, allowing one mole to be collected in 50.4 minutes. The flow of air through the leak is turned on or off with a remotely commanded, pneumatically operated, high-vacuum valve (Varian P/N 951-5090) fitted with a magnetically latching dc solenoid and using compressed helium as the working gas.

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1. Gould, P.R. (1974) Cryogenic Whole Air Sampler System, Tech Report AFCRL-TR-74-0452.
2. Dushman and Laferty (1962) Scientific Formulations of Vacuum Technique, p. 82, J. Wiley and Co.

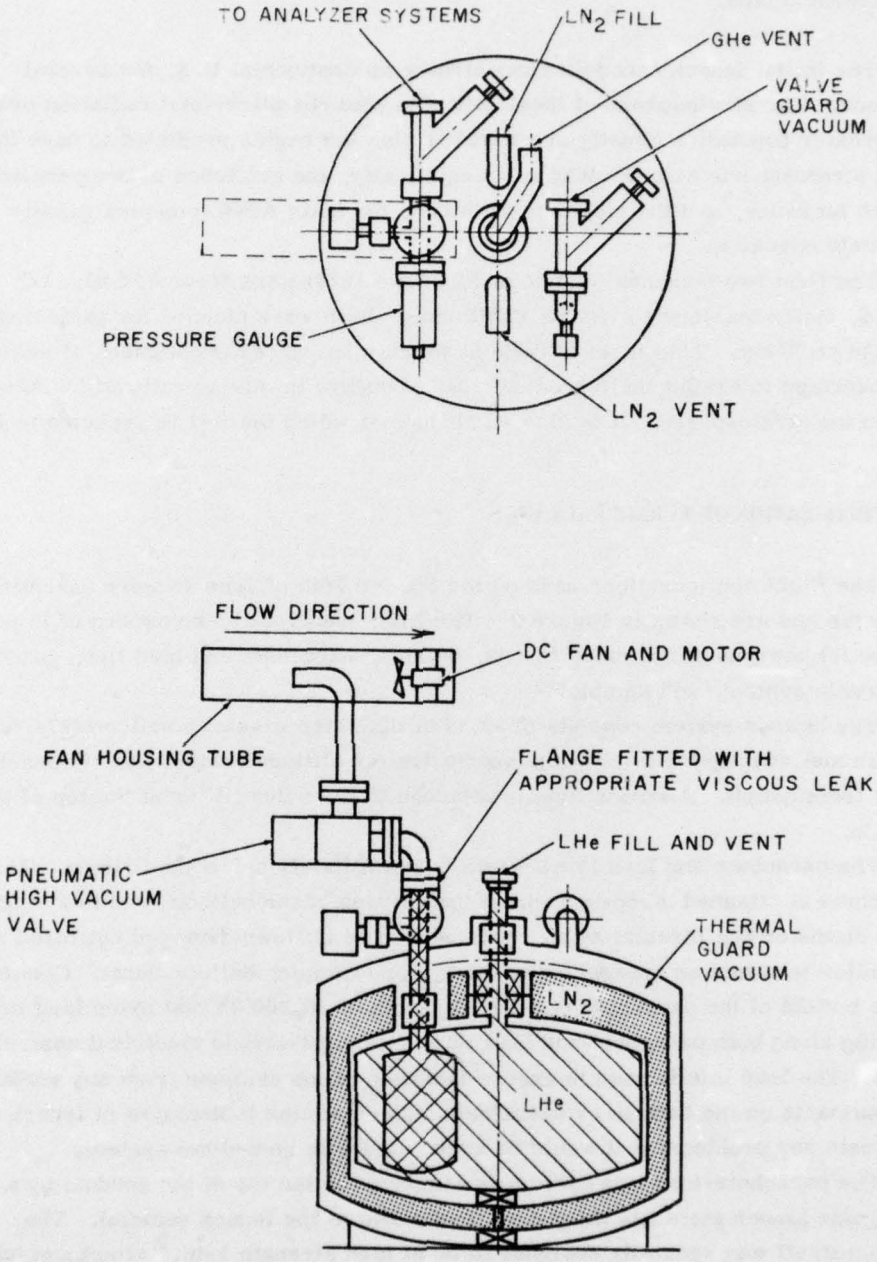

Figure 1. Sampler and Air Intake System

5. FY75 LAUNCHES

The initial launch schedule concentrates on continental U. S. for several reasons. The stratosphere at these latitudes absorbs ultraviolet radiation over the greater population density areas and is also the region predicted to have the most stratospheric aircraft traffic. Logistically, the existence of two permanent launch facilities, and the closer proximity to the main AFGL complex greatly facilitate operation.

The first two launches (5 June and 26 June 1975) were from AFCRL, LC, Det. 3, Chico Municipal Airport, California. Both were planned for gathering a sample at 20 km. This is an altitude at which many of the trace gases of interest are expected to exhibit their greatest rate of change in mixing ratio with altitude within the stratosphere. It is also an altitude at which the B-1 is expected to fly.

6. SPECIFICATION OF FLIGHT PACKAGE

The flight configurations used on the 5th and 26th of June 75 were essentially the same and are shown in Figure 2. The flight package can be spoken of in terms of five (5) component systems, that is, balloon, parachute and load line, gondola, electronic control, and sampler.

The balloon system consists of an 87-ft diameter research balloon (274, 000-ft^3 volume) equipped with a helium vent valve for altitude control and rip panels for flight termination. A strobe light is attached to the valve fitting at the top of the balloon.

The parachute and load line system is immediately below the balloon. The parachute is attached directly to the bottom fitting of the balloon, and is a single 48-ft diameter flat circular type. This parachute is flown deployed but fitted with a monitor to detect an unexpected opening, for example, balloon burst. Connected to the bottom of the parachute is a 200-ft length of 10, 000-lb test nylon load line. Running along both parachute and load line is an eight-strand electrical control cable. The load line is used to ensure isolation of the sampler from any surface contaminants on the balloon or parachute. The load line is deployed at launch to eliminate any problems that would be associated with reel-down systems.

The parachute-load line system is connected to the top of the gondola by a triangular launch plate (its third corner attaches to the launch vehicle). The gondola itself was specially designed to be of high strength 1-$in.^2$ aluminum tubing. It is spherical, except for the base, and is 87 in. in diameter, with equipment mounting provisions such that when loaded its center of gravity is as low as possible. This design was intended to make the gondola self righting. To remove any

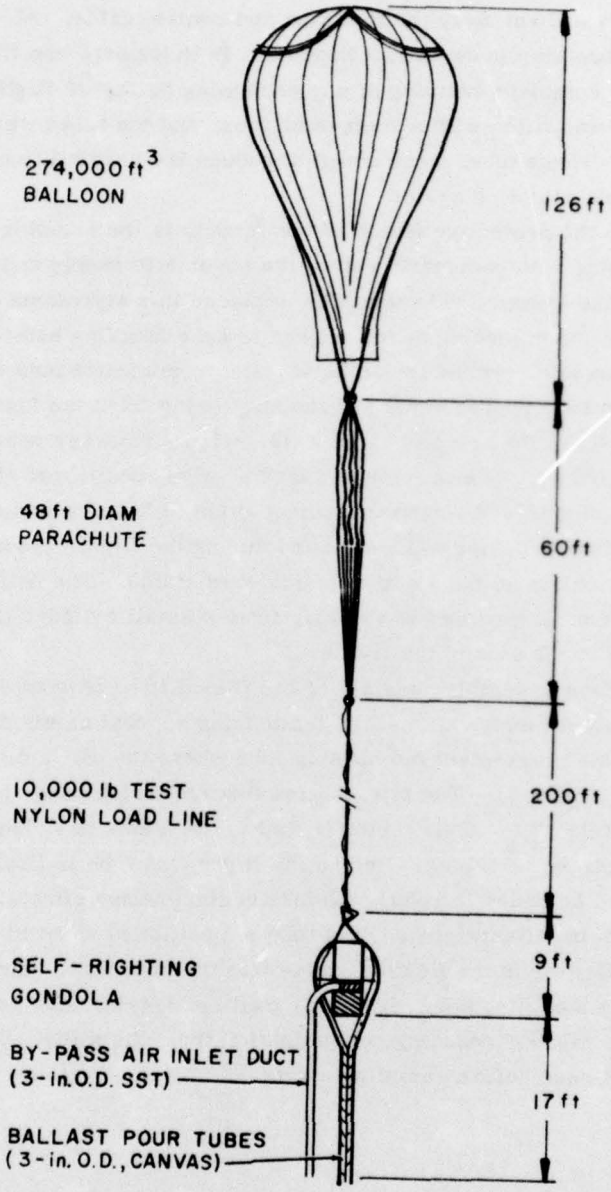

Figure 2. Flight Package

possibility of the gondola becoming entangled in the load line or being dragged by the parachute, the gondola is equipped with four impact switches that, upon landing, fire a pair of squibs and cut away the load line and control cable. Also attached to the gondola are two aluminum ballast hoppers. Both hoppers are fitted with blow ports to allow complete dumping of any remaining ballast at flight termination. Each hopper was fitted with a 20-ft cloth tube, and the tubes were joined below the gondola. These tubes were added to reduce the possibility of any ballast being ingested by the sampling system.

Mounted within the protective frame of the gondola is the sampler system consisting of the sampler with pneumatic valve, the pneumatic supply and control, and the sample intake system. The sampler is placed in a styrofoam box (2 in. thick) to reduce the shock loading at impact and to ease handling before launch. The sampler and box are covered by a plastic sheet to minimize both the condensation which forms on the cryogen vents and the outgassing from the foam from affecting the integrity of the sample. Five 2-in. nylon straps are used to secure the sampler to the frame. The pneumatic vacuum valve, positioned above the plastic sheet, is equipped with a heater and insulation to keep its sealing gasket from freezing and then cracking when operated during the flight. Added for the second flight is a monitor on the valve to indicate its status. The helium used in the pneumatic systems is supplied at 100 psig from a small cylinder (32 ft^3 at 1900 psig) attached to the side of the frame.

The sample intake assembly consists of two joined 10-ft lengths of 3-in. OD flexible stainless-steel tubing, a small dc fan moving approximately 40 ft^3 of air per minute, and a stainless-steel fan housing tube where the air is diverted to the vacuum valve (see Figure 1). The inlet for the flexible tubing hangs below the gondola approximately 17 ft. This tubing is tied to the frame to reduce any damage resulting from a pull on the tubing. Before the flight, the tube is flushed with helium gas for 18 to 36 hours to reduce surface contamination effects. In the rigid housing tube, a 3/4-in. OD stainless-steel tube is positioned so as to divert the center plug of air flowing in the flexible tubing into the pneumatic valve. The fan, mounted behind this sampling port, draws air past the 3/4-in. tube so that the flow regime is laminar, thereby reducing contamination from the walls. The fan is turned on about 1/4 hour before sampling starts.

7. ELECTRONICS

All power, command, control, and data gathering capabilities were maintained in the electronic system supplied by the Aerospace Instrumentation Laboratory at AFGL. The electronic system was specially designed for this mission, however,

the package will only be functionally described here. The unit basically contains battery supplies, HF command receiver, command decoder, HF data transmitter, data encoder, flight safety timing, logic control unit, impact switch control unit, pressure and temperature sensors. There is also a separate back-up package which provided command redundancy for the more important control functions. The HF command receiver works in conjunction with the decoder and finally with the logic control unit which energizes all control functions and sequences. Upon channel acquisition, the decoder generates a code which interrupts the normal data cycle and this code is transmitted to the ground control station. The coded reply tells the ground operation that the proper channel has been selected and guarantees the correct activation of the selected channel by the energize command. (Nine channels are available in the main package and six in the back-up package.) The encoder converts a 0 to 5.1-V dc input signal into a 9-bit octal code which is transmitted to the ground station where it is recorded and decoded. The accuracy of the encoder is ±5 mV which corresponds to ±60 ft in the sampling region of both balloon flights. (Seven channels and four subcommutated channels are available.) A combination of three pressure sensors is used for each flight to maintain a good pressure vs altitude resolution. All pressure sensors are carefully calibrated and cross referenced to a standard altitude-pressure profile.

8. METHOD, COMMAND, AND MONITORING

In pre-flight planning discussions it was decided to monitor several aspects of the flight, primarily for post-flight analysis in the event a problem developed. As mentioned previously, the electronic control unit is designed so that the execution of a particular command can be confirmed through the telemetry link with the payload. This characteristic is used to confirm that the three primary or the three back-up commands, that is, Fan On, Valve Open and Fan Off - Valve Close, are transmitted through the electronic system and are being acted on by the sampler package. The voltages applied to the fan's motors and to the solenoid controlling the pneumatic valve are also monitored and reported back to the ground station. Five other parameters are recorded. These parameters are: the level of liquid in the sampler and four temperature readings taken at: the liquid helium boiloff vent, the liquid nitrogen boiloff vent, the surface of the sampler (away from the vents), and the surface of the gondola. A position monitor was used on the pneumatic valve for the June 26th flight.

The liquid helium level sensor is based on the characteristic rapid increase in resistance shown by Allen-Bradley carbon composition resistors as they approach the temperature of liquid helium. The sensing probe is composed of three resistors

placed at the 2-1/2, 8-1/2, and 13-1/2 liter levels in the liquid helium dewar. The sensitivity circuit is designed to use as little power as possible (milliwatts) to detect the resistance change. From the value of each resistor, at each moment, the circuit forms a single unit of data, reflecting the amount of liquid helium remaining in the dewar.

The reason for monitoring the temperature of the cryogenic vents and the surrounding hardware was to determine the cryogenic boiloff rates. Any temperature differences would be due to cooling caused by the venting gas and we would be able to correlate the boiloff rates with the temperature differentials. The temperature sensors used were two thermistors and two silicon unijunction transistors. Ambient temperature and pressure measurements are also available from radiosonde units attached to the gondola for each flight.

The position indicator used with the pneumatic valve provides the ground station with positive confirmation that the vacuum sampling system is open. The position indicator involves two relay switches which take advantage of the magnetic qualities of the plunger of the pneumatic valve. They are positioned near the limits of travel in such a manner as to detect: closed, half open, and fully open positions. The greatest use made of the sensors was for inflight decision making. The sensors are also important indicators of certain conditions which would cause abortion of the mission.

Excluding minor problems, that is, apparent thermal opening of a joint on one of the unijunction transistors, the only major difficulty with the sensing systems was that the helium level indicator would become unstable during periods of sampling, probably due to the rapid boiloff and resulting agitation of liquid. This problem would disappear after sampling, but would reappear to a much lesser degree during descent on the parachute.

9. 20-km FLIGHT OF 5 JUNE 1975

The first launch within the Stratospheric Composition Program was conducted at Chico (approx. 40°N lat) and designated AFCRL launch C75-10. The desired sampling altitude was 20 km, and it would take 50 minutes to capture and freeze 1 mole at the 40-mm ambient pressure.

Launch was scheduled for shortly after dawn because, statistically, winds at the launch site are lowest at this time and the descent and impact would still occur prior to the afternoon increase in wind speeds. Also, the stratospheric NO concentration would have recovered from its nighttime minimum by sampling time.

The sampler is maintained at liquid cryogen temperatures for a minimum of 12 hours prior to flight. Immediate pre-flight preparations consume about 5 hours

and include "topping off" the sampler cryogens, testing command and telemetry electronics and, of course, preparation of the balloon launch package.

Launch took place at 0640 PDT and the balloon ascended to the southeast, reaching its float altitude of 67, 100 ft at 0808 PDT.

This altitude would allow the requirement to be fulfilled that sampling be limited to a descending mode within 20 ± . 5 km (65, 600 ± 1600 ft). Sampling during descent minimizes the chances of any outgassing from the gondola package or balloon ever reaching the sampling tube intake. The ±. 5-km tolerance pinpoints the data to a much more accurate altitude specification than is possible with alternate measurement techniques.

With the specified sampling time of 50 minutes, a descent rate of 50 ft/min was desired to stay within the sampling altitude range. This was achieved through balloon valving and gondola ballasting.

The desired air flow was established in the tube and sampling commenced at 0901 PDT. Two brief interruptions in sampling were required for adjustment of the descent rate, and sampling was completed at 1015 PDT.

Subsequently, additional valving and ballasting were accomplished to place the balloon in an airstream that would bring the balloon into a favorable position near Chico for termination. Then, the package was commanded to descend on parachute and it landed at a 45° angle in brush, undamaged but in an unexpected and inaccessible location that required helicopter retrieval. Fortunately, sufficient cryogens still remained upon arrival back in Chico for preservation of the air sample.

10. 20-km FLIGHT OF 26 JUNE 1975

The second launch (AFCRL Launch C75-11) took place on 26 June at Chico. The desired sampling altitude was 20 km and the specified sampling time was again 50 minutes.

The preparations and launch day schedule were similar to those for the first launch. The launch took place at 0715 PDT and the balloon ascended towards the east, reaching float altitude in 1-1/4 hours.

The specified descent rate was achieved by valving and ballasting and the 50-minute sampling period was completed by 1000 PDT. The average descent rate was about 20 ft/min and so the sample was gathered over a narrower altitude increment than on the first flight.

Following sampling, the balloon was directed to the northern end of the Sacramento Valley where the terrain was much more suited for a smooth landing and recovery. Termination and parachute descent were normal and, upon impact, the package rolled several times down an incline, landing in an upright position

against a tree. The package was recovered 35 minutes later undamaged and with the sample intact.

11. DIAGNOSTICS

Three diagnostic systems are utilized for analysis of air samples. A portion of the whole-air sample is analyzed directly. The remainder of the sample is fractionated with the aid of a cryogenic still to provide three fractions: one high, one medium, and one low boiling point sample. The fractionation facilitates analysis by providing less gas species per sample, in particular, it separates off the dominant oxygen and nitrogen components, which easily saturate some diagnostic systems.

An NO-NO_x chemiluminescent analyzer, a gas chromatograph, and a mass spectrograph are used to analyze samples.

The chemiluminescent analyzer, a Thermo Electron Corp., Model 12A modified with the incorporation of a Keithley Model 640 vibrating reed electrometer, permits NO and NO_x measurements down to less than 1 ppb concentration. This device provides the quickest analysis and is used on the whole-air sample immediately after sample warmup and simultaneous expansion into a large vacuum vessel. In this way, the NO and NO_2 concentrations are measured before any significant quantity of chemical changes affecting these more reactive species can occur.

The gas chromatograph is a Varian Aerograph Trace Gas Analyzer, Model 2732, a dual column instrument with helium ionization detectors (250 mc ^3H) and a dual differential electrometer. The practical limit of detection is 2 ppb (that is, for nitrogen and carbon monoxide but not for all gases).

Although the expected range of concentrations of some of the trace gases in the whole-air sample lie below these detection limits, the enrichment that occurs in the medium- and high-boiling point fractionated samples results in all gases of interest being present in concentrations above the detection thresholds. The chromatograph will be modified to provide even greater sensitivity.

Both 3-ft and 6-ft molecular sieves and Porapak Q columns are used. Porasil A and other packing materials would be used later to enhance elution peak separations and improve the accuracy of the quantization of the various output peaks. Recording and integration are provided by a Hewlett Package Model 3380A Integrator.

The third analysis instrument is an Extranuclear Laboratories Quadrupole Mass Spectrometer. This mass filter is set up for use with positive ions, utilizing electron impact ionization. The electron multiplier is a 14-stage "venetian blind" type mounted with its axis parallel to the center axis of the rods. The electrometer used with this system was developed by the Composition Branch for use on experimental rocket-borne quadrupole filters. The electrometer is logarithmic from

2×10^{-5}A to 1×10^{-12}A with a minimum of about 20 msec response time. The spectrometer is instrumented to scan from 0 to 250 amu in approximately 25 sec and has the capability of being programmed to study several particular mass units for time averaging work. The present sensitivity obtained with the instrument is 74 mA torr for N_2, with a resolution of approximately 1 amu at 2×10^{-5} torr.

12. CONCLUSIONS AND ASSESSMENTS

The goal of capturing and freezing 1 mole of stratospheric air and returning it in its frozen state to the laboratory was accomplished.

This success has demonstrated that even under the severe conditions of flight, cryogen losses are not so great as to result in loss of the sample and the sampler can survive the heavy mechanical forces of impact. For the sampling orifice chosen, the sampling time produced approximately the sample quantity predicted.

The remotely controlled, pneumatically activated valve responded satisfactorily to commands and sealed tightly. The gondola design afforded convenient access to equipment and provided adequate protection upon impact. The roll qualities, designed to ensure an upright rest position following impact, were not sufficiently tested as neither impact occurred in open country. The various temperature and cryogen-level monitoring signals were received as desired. The remainder of the standard flight package components (balloon, and so forth) and the commands and telemetry operated as required.

The chief problem was to get the sample back to the laboratory while still frozen. Even with an experimenter accompanying the sample, the pilots showed an aversion to carrying a container of cryogens.

13. PROJECTIONS

The success with the sampler and overall flight package on the first two flights suggested that the same configuration and operations be used on succeeding flights with just a few changes. Some adjustments to the command and control features were expected to further increase operational reliability and sample integrity.

Specifically, for FY'76, the air intake system will be modified so that a constant air stream would be maintained through the body of the valve. Also, a motor activated valve employing a metal to metal seal will be substituted for the pneumatically operated valve which contained a non-metallic seal. There will be a heavier flight schedule including expansion to other of the prescribed altitudes and latitudes. Sampling tubes for the other altitudes will be designed on the same principle as that used for the 20-km flights.

For FY'77 flights, two tri-samplers will be designed and fabricated, with each such unit containing three sample chambers and controls, thereby permitting sampling at three altitudes on one flight. In addition to the obvious advantages of more efficient, economical operation, the tri-sampler, by obtaining three samples almost simultaneously, will afford a better opportunity to compare data from different altitudes without the temporal effects that might arise when samples at each altitude are obtained on separate days.